A Novel Hybrid Ultramicrotomy/FIB-SEM Technique: Preparation of Serial Electron-Transparent Thin Sections of a Hayabusa Grain.

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The Japanese space agency's (JAXA) Hayabusa mission returned the first particulate samples (typically <100μm) from the surface of an asteroid (25143 Itokawa). These precious samples provide important insights into early Solar System processes, but their sizes pose tremendous challenges to coordinated analysis using a variety of nano- and micro-beam techniques. The ability to glean maximal information from individual particles has become increasingly important and depends critically on sample preparation. We developed a hybrid technique combining traditional ultramicrotomy with focused ion beam (FIB) techniques, allowing for more thorough *in situ* investigations of grain surfaces and interiors. Using this method, we increase the number of FIB-prepared sections that can be recovered from a particle with dimensions on the order of tens of microns. These sections can be subsequently analyzed using a variety of analytical techniques.

Particle RA-QD02-0211 is a ~40×40×20μm particle from Itokawa containing olivine and Fe sulfides. It was embedded in low viscosity epoxy and partly sectioned to a depth of ~10μm; sections are placed on Cu grids with thin amorphous films for transmission electron microscope (TEM) analyses. With the sample surface partly exposed, the epoxy bullet is trimmed to a height of ~5mm to accommodate the allowable dimensions for FIB work (FEI Quanta 600 3D dual beam FIB-SEM). Using a diamond trim knife, the epoxy surrounding the grain is removed on 3 sides (to within a few microns of the grain); the depth of material removed extends well below the bottom of the particle (fig. 1). The sample is attached to an SEM pin mount, the epoxy coated with conductive paint, and the entire assembly coated with ~40nm of carbon to eliminate sample charging during FIB work.

A protective carbon cap is placed according to the plan for the 15 FIB sections (fig. 2). The central 'spine' of the cap runs perpendicular to the front of the sample, and the 'ribs' protruding from either side run parallel. Each rib indicates the location of a planned FIB section, and the spine contains the final two planned sections. We use a cap with a 4μm-wide spine and 2μm-wide ribs that have ≳3.5μm of space between them (narrower cuts result in too much re-deposition of material inside the trenches). Using a 30kV, 3nA ion-beam we expose the front surface of the grain and commence milling trenches between sections (fig. 2). Rather than using the typical C-cut to prepare the sample for lift-out, an L-cut is used instead, leaving the sample connected by an interior tab. Sections are lifted out, attached to TEM grids and thinned to electron transparency.

TEM analyses show that our hybrid technique preserves both interior and edge features, including surface modifications from exposure to the space environment, such as damaged rims that form in response to solar wind implantation effects and adhering grains. In addition, the FIB sections provide larger areas that are free of fractures and chatter effects in comparison to the microtome thin sections, thus enabling more accurate measurements of solar flare particle track densities (fig. 3) that are used to determine the surface exposure age of the particles.

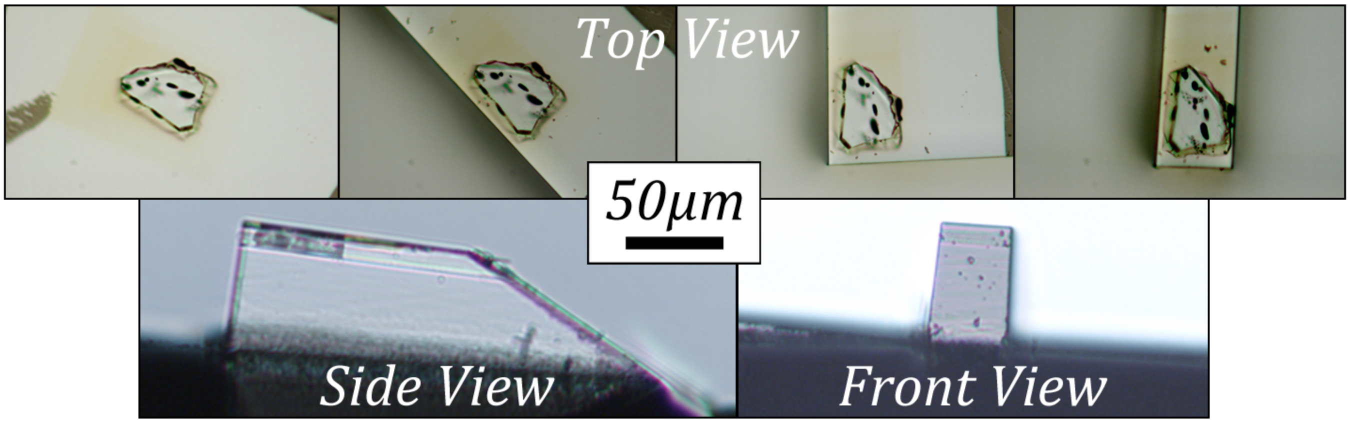

Figure 1. Reflected and transmitted light images of the grain embedded in epoxy and partly sectioned. After sectioning, the epoxy surrounding the grain is trimmed away on three sides.

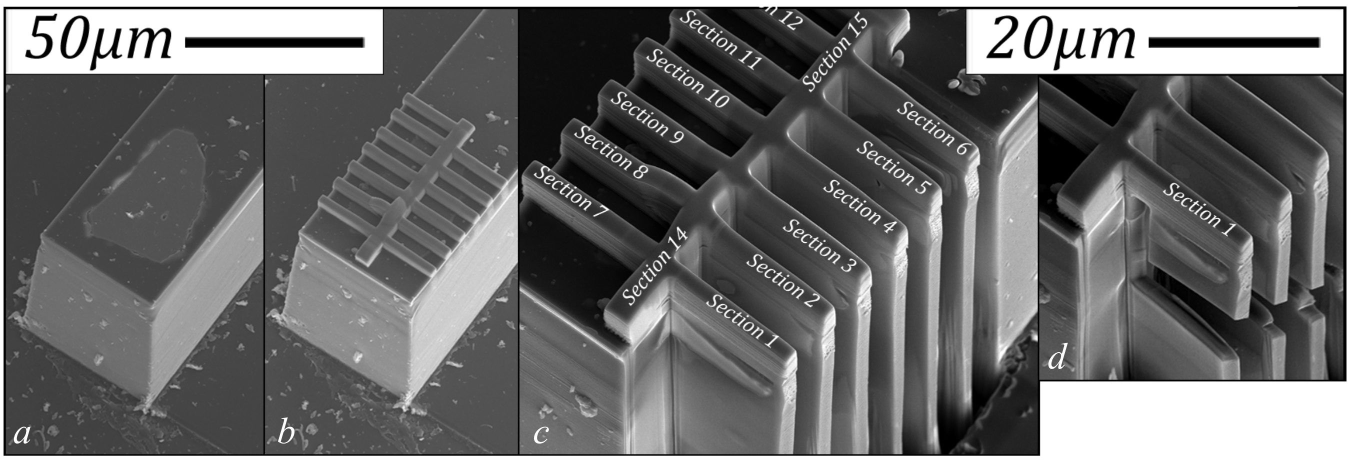

Figure 2. Secondary electron images of the grain from an oblique angle. (*a*) The exposed grain is at the end of a rectangular box of epoxy, sitting on the bulk of the substrate. (*b*) The protective carbon cap is placed over the areas of interest. (*c*) The locations of the 15 planned FIB-prepared electron transparent thin sections are indicated. Material between sections 1 through 6 is milled away. (*d*) An L-cut is made to remove material under section 1 in preparation for lift-out and thinning.

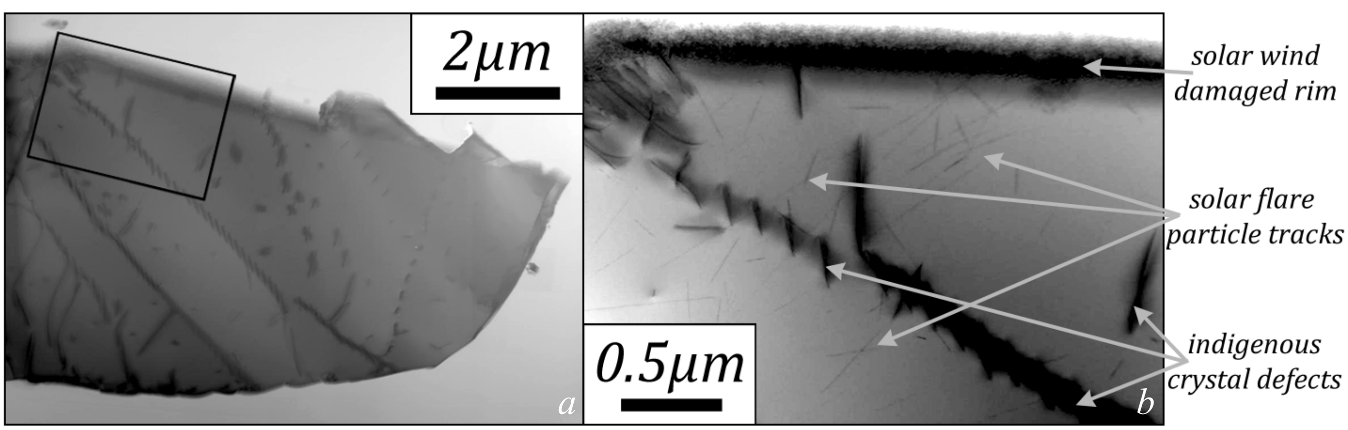

Figure 3. Bright-field images (BFIs) of section 1, after being lifted out and thinned to ~100−150nm. Features of space weathering (solar flare particle tracks, disordered rims, and nanophase particles decorating the outer edges) can be seen in the BFIs. (*a*) The entire section surrounded by epoxy; The C-cap is above the field of view, the TEM grid bar to the left. (*b*) Close-up of the area indicated in panel a.